MEMORANDUM

TO: Mr. Addison Rice

DATE: June 28, 2016

Anderson, Malholland and Associates

FROM: R. Infante

FILE: 1605308CR1

RE:

Data Validation Air samples

SDG: 1605308CR1

SUMMARY

Full validation was performed on the data for several air samples analyzed for methane by ASTM method D-1946-modified. The samples were collected at the Bristol Myer Squib facility, Humacao, PR site on May 14, 2016 and submitted to Eurofins Air Toxics, Inc. of Folson, California that analyzed and reported the results under delivery groups (SDG) 1605308CR1.

The sample results were assessed according to USEPA data validation guidance documents in the following order of precedence: Volatile Organic Analysis of Ambient Air in Canisters by Method TO-15, (SOP # HW-31. Revision #4. October, 2006; and the QC criteria of the ASTM method D-1946-modified. The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

In general the data is valid as reported and may be used for decision making purposes. The data results are acceptable for use.

SAMPLES

The samples included in the review are listed below

Client Sample ID	Lab. Sample ID	Collected Date	Matrix	Analysis
B71A-1D	1605308CR1-02A	05/14/2016	Air	Methane
B71A-3	1605308CR1-04A	05/14/2016	Air	Methane
B71A-4	1605308CR1-05A	05/14/2016	Air	Methane
B71A-5	1605308CR1-06A	05/14/2016	Air	Methane

REVIEW ELEMENTS

Sample data were reviewed for the following parameters, where applicable to the method

- o Agreement of analysis conducted with chain of custody (COC) form
- o Holding time and sample preservation
- o Gas chromatography/mass spectrometry (GC/MS) tunes
- Initial and continuing calibrations
- Method blanks/trip blanks/field blank
- o Canister cleaning certification criteria

- Surrogate spike recovery
- o Internal standard performance and retention times
- o Field duplicate results
- o Laboratory control sample/laboratory control sample duplicate (LCS/LCSD) results
- o Quantitation limits and sample results

DISCUSSION

Agreement of Analysis Conducted with COC Request

Sample reports corresponded to the analytical request designated on the chain-of-custody.

The Chain of Custody (COC) information for sample B7IA-1D did not match the information on the canister with regard to canister identification. The client was notified of the discrepancy and the information on the canister was used to process and report the sample.

Holding Times and Sample Preservation

All samples analyzed within the recommended method holding time. All summa canisters received in good conditions.

Samples analyzed within method recommended holding time.

Initial and Continuing Calibrations

Methane by ASTM method D-1946 (modified)

Initial and continuing calibrations meet method specific requirements. Initial calibration retention times meet method specific requirements.

Method Blank/Trip Blank/Field Blank

Target analytes were not detected in laboratory method blanks.

No trip/field blank analyzed with this data package.

Laboratory/Field Duplicate Results

Laboratory duplicates were analyzed as part of this data set. Target analytes meet the RPD performance criteria of +25% for analytes $5\times SQL$.

LCS/LCSD Results

<u>Methane</u>

LCS/LCSD (blank spike) were analyzed by the laboratory associated with this data package. Recoveries and RPD within laboratory control limits.

Quantitation Limits and Sample Results

Dilutions were not performed (see worksheet).

Calculations were spot checked.

Certification

The following samples 1605308C-02A; 1605308C-04; 1605308C-05A; and 1605308C-06A were analyzed following standard procedures accepted by regulatory agencies. The quality control requirements met the methods criteria except in the occasions described in this document.

Rafael Infante

Chemist License 1888



Client Sample ID: B7IA-1D Lab ID#: 1605308CR1-02A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name: Dil. Factor:	10051713 1.66	Date of Collection: 5/14/16 8:12:00 F Date of Analysis: 5/17/16 02:24 PM	
Compound		Rpt Limit (%)	Amount (%)
Methane		0.00017	0.00017





Client Sample ID: B7IA-3 Lab ID#: 1605308CR1-04A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name:	10051714	Date of Collect	tion: 5/14/16 8:34:00 PM
Dil. Factor:	1.80	Date of Analysis: 5/17/16 02:46 PM	
		Rpt. Limit	Amount
Compound		(%)	
Methane		0.00018	0.00016 J

J = Estimated value.





Client Sample ID: B7IA-4 Lab ID#: 1605308CR1-05A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name: Dil. Factor:	10051715 1.62		tion: 5/14/16 8:57:00 PM
		Rpt. Limit	Amount
Compound		(%)	(%)
Methane	···	0.00016	0.00017



Air Toxics

Client Sample ID: B7IA-5 Lab ID#: 1605308CR1-06A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name: Dil. Factor:	10051716 1.58	Date of Collection: 5/14/16 8:04:00 PM Date of Analysis: 5/17/16 03:33 PM	
Compound		Rpt. Limit (%)	Amount (%)
Methane		0.00016	0.00019



eurofins

Sample Transportation Notice

Relinquishing signature on this document indicates that sample is being shipped in compliance with 180 BLUE RAVINE ROAD, SUITE B all applicable local, State, Federal, national, and international laws, regulations and ordinances of any kind. Air Toxics Limited assumes no liability with respect to the collection, handling or shipping

Fedex tracking No. 7830 81486749

of these samples. Relinquishing signature also indicates agreement to hold harmless, defend, and indemnify Air Toxics Limited against any claim, demand, or action, of any kind, related to the

FOLSOM, CA 95630-4719 (916) 985-1000 FAX (916) 985-1020

	and indemnity a collection, hank	Air Toxics Limited Iling, or shipping o	against any clai if samples. D.O	m, demand, or act T. Hotline (800) 46	ion, of any kind, related to th i7-4922	ne	Pa	ge	of
	Project Manager Terry Laylor Collected by: (Print and Sign) Terry Taylor + David L	Later and	Proj	ect Info:		Turn Around		<i>Only</i> urized by:	
	Company Anderson Mulholland Asstanail Trays		P.O.			☐ Normal	Date		
	Address 2700 West checter City Purchase State	NY Zin M	Proje	ct #		⊠ (Rush	140000	unzation (Gaa-
	Phone 914 -251 -0400 Fax	— t		ct Name		1-Day		N ₂ H	
			Date	Time		specify/	ter Pres	sure/Vac	
	Lib I.D. Field Sample I.D. (Location)	Can #		of Collection	Analyses Reques		Final		
	B7 IA-1	06300	05/14/16	2011	10-15	+30	7.5	4 32 3 4 5 4	(påi)
Į¢Κ	B7 IA-ID	00355	05/14/16	2012	T0-15	+30	7.0		41.0
5/11/1	B7 IA-2	N0593	1,1,1,1	2025	To-15	130	13.0	a secondario	
	010 B7 IA-3	00314	05/14/16	2034	10-15	+ 30	10.0	4	
	105a B7 IA-4	5737	05/14/16	2057	TO-15	+30	6.5	West of	
	B7 IA-5	6L1224	05/A/16		10-15	+30	6.5	(Q. 1)	
KK	67 IA - 6	00316	05/14/16	2053	T0-15	+30	~17		, 41
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	AND M								<i>'</i> 5
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	Use Grapy	AND ROWSELL BASE AND MAKE A RECOGNISHED STATES.	#mp (°C)	Condition			PROPERTY AND	Order #	
	Only	/v	//	6000	Yes No	(None	1	67530	18_

	Date:05/14/2016
	00/11/2010
REVIEW OF VOLATILE ORGATINE The following guidelines for evaluating volatile organics was actions. This document will assist the reviewer in using prodecision and in better serving the needs of the data users. The USEPA data validation guidance documents in the following D-1946 method for measuring permanent gases and light samples using gas chromatography (GC) and a thermal conditional detection (FID). Validating Air Samples. Volatile Organic And TO-15, (SOP # HW-31. Revision #4. October, 2006). The QC the data review worksheets are from the primary guidance documents and the quality control and performance data summare.	ere created to delineate required validation of pressional judgment to make more informed the sample results were assessed according to order of precedence: QC criteria from ASTM hydrocarbons in refinery and other sources uctivity detector (TCD) and/or flame ionization alysis of Ambient Air in Canisters by Method C criteria and data validation actions listed on nument, unless otherwise noted. data package received has been
Lab. Project/SDG No.:1605308CR1 No. of Samples:4	Sample matrix:Air
Trip blank No.: Field blank No.: Equipment blank No.: Field duplicate No.:	
X Data CompletenessX Holding TimesN/A_ GC/MS TuningN/A_ Internal Standard PerformanceX BlanksN/A_ Surrogate RecoveriesN/A_ Matrix Spike/Matrix Spike Duplicate	X Laboratory Control SpikesX Field DuplicatesX CalibrationsX Compound IdentificationsX Compound QuantitationX Quantitation Limits
Overall Comments:_Methane_by_ASTM_method_D-194	6_(modified)
Definition of Qualifiers: J- Estimated results U- Compound not detected R- Rejected data UJ- Estimated nondetect Reviewer:	
Date: 06/28/2016	

DATA REVIEW WORKSHEETS

DATA COMPLETENESS

MISSING INFORMATION	DATE LAB. CONTACTED	DATE RECEIVED
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	<u> </u>	-
	1820.	
	1	
	1	
S 12 - 2000 A 10 A	\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\\	
		*

All criteria were met _	X_	_
Criteria were not met		
and/or see below		

HOLDING TIMES

The objective of this parameter is to ascertain the validity of the results based on the holding time of the sample from time of collection to the time of analysis.

Complete table for all samples and note the analysis and/or preservation not within criteria

SAMPLE ID	DATE SAMPLED	DATE ANALYZED	рН	ACTION
			+	
All samples analy:	zed within the recomm	nended method holding	time. All	summa canisters received
				B7IA-1D did not match the
		to canister identification		
discrepancy and ti	he information on the	canister was used to pi	ocess a	nd report the sample.
				On a
	+		+	

<u>Criteria</u>

Aqueous samples – 14 days from sample collection for preserved samples (pH \leq 2, 4°C), no air bubbles.

Aqueous samples – 7 days from sample collection for unpreserved samples, 4°C, no air bubbles. Soil samples- 7 days from sample collection.

Cooler temperature (Criteria: 4 ± 2 °C): N/A – summa canisters

<u>Actions</u>

If the VOCs vial(s) have air bubbles, estimate positive results (J) and reject nondetects (R).

If the % solids of soil samples is 10-50%, estimates positive results (J) and nondetects (UJ)

If the % solid of soil samples is < 10%, estimate positive results (J) and reject nondetects (R).

If holding times are exceeded but < 14 days beyond criteria, estimate positive results (J) and nondetects (UJ).

If holding times are exceeded but < 28 days beyond criteria, estimate positive results (J) and reject nondetects (R).

If holding times are grossly exceeded (> 28 days beyond criteria), reject all results (R).

If samples were not iced or if the ice were melted (> 10°C), estimate positive results (J) and nondetects (UJ).

DATA REVIEW WORKSHEETS

qualified or rejected.

All criteria were metN/A_ Criteria were not met see below	
GC/MS TUNING	
The assessment of the tuning results is to determine if the sample instrumentation is within the standard tuning QC limits	1
N/A_ The BFB performance results were reviewed and found to be within the specified criteria.	
N/A_ BFB tuning was performed for every 24 hours of sample analysis.	
If no, use professional judgment to determine whether the associated data should be accepted,	

List

the

samples

affected:

If mass calibration is in error, all associated data are rejected.

Note: Samples analyzed using GC with either TCD or FID detection.

All criteria were met _X
Criteria were not met
and/or see below

01/15/16

CALIBRATION VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration:

				Dates of continuing calibra Instrument ID numbers:Matrix/Level:	GC-10
DATE	LAB ID#	FILE	CRITERIA OUT RFs, %RSD, %D, r	COMPOUND	SAMPLES AFFECTED
		_	rations meet method requirements.	specific requirements. Init	ial calibration retention
	1			l l	I .

Criteria

All RFs must be > 0.05 regardless of method requirements for SPCC.

All %RSD must be < 15 % regardless of method requirements for CCC.

All %Ds must be ≤ 30% regardless of method requirements for CCC.

Method TO-15 does not specify criterion for the curve correlation coefficient (r). A limit for r of \geq 0.995 has therefore been utilized as professional judgment.

Actions

If any compound has an initial RF or a continuing RF of < 0.05, estimate positive results (J) and reject nondetects (R), regardless of method requirements.

If any compound has a %RSD > 15%, estimate positive results (J) and use professional judgment to qualify nondetects.

If any compound has a %RSD > 90%, estimate positive results (J) and reject nondetects (R).

If any compound has a % D > 30%, estimate positive results (J) and reject nondetects (R).

If any compound has a % D > 30%, estimate positive results (J) and nondetects (UJ).

If any compound has a % D > 90%, estimate positive results (J) and reject nondetects (R).

If any compound has r < 0.995, estimate positive results and nondetects.

A separate worksheet should be filled for each initial curve

All criteria were met _X
Criteria were not met
and/or see below

V A. BLANK ANALYSIS RESULTS (Sections 1 & 2)

The assessment of the blank analysis results is to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks apply only to blanks associated with the samples, including trip, equipment, and laboratory blanks. If problems with any blanks exist, all data associated with the case must be carefully evaluated to determine whether or not there is an inherent variability in the data for the case, or if the problem is an isolated occurrence not affecting other data.

List the contamination in the blanks below. High and low levels blanks must be treated separately.

Laboratory blanks

DATE ANALYZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS
All_metho	_	- 443	fic_criteria	
Field <u>/</u> Equipmen				
DATE Analyzed	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS
_No_field/trip/e	quipment_blank	s_analyzed_wi	th_this_data_package	
			1. 4.0	

All criteria were met _	х_
Criteria were not met	
and/or see below	

VB. BLANK ANALYSIS RESULTS (Section 3)

Blank Actions

Action Levels (ALs) should be based upon the highest concentration of contaminant determined in any blank. Do not qualify any blank with another blank. The ALs for samples which have been diluted should be corrected for the sample dilution factor and/or % moisture, where applicable. No positive sample results should be reported unless the concentration of the compound in the samples exceeds the ALs:

ALs = 10x the amount of common contaminants (methylene chloride, acetone, 2-butanone, and toluene)

ALs = 5x for any other compounds

Specific actions are as follows:

If the concentration is < sample quantitation limit (SQL) and \le AL, report the compound as not detected (U) at the SQL.

if the concentration is \geq SQL but \leq AL, report the compound as not detected (U) at the reported concentration.

If the concentration is \geq SQL and > AL, report the concentration unqualified.

Notes:

High and low level blanks must be treated separately

Compounds qualified "U" for blank contamination are still considered "hits" when qualifying for calibration criteria.

CONTAMINATION SOURCE/LEVEL	COMPOUND	CONC/UNITS	AL/UNITS	SQL	AFFECTED SAMPLES
					- Hillians
					MITTER STATES
				eds.	
			32		
		-115			
	10A		İ		
1	1				
-					

All criteria were met __N/A__ Criteria were not met and/or see below ____

ACTION

SURROGATE SPIKE RECOVERIES

Laboratory performance of individual samples is established by evaluation of surrogate spike recoveries. All samples are spiked with surrogate compounds prior to sample analysis. The accuracy of the analysis is measured by the surrogate percent recovery. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the validation of data is frequently subjective and demands analytical experience and professional judgment.

SURROGATE COMPOUND

List the percent recoveries (%Rs) which do not meet the criteria for surrogate recovery.

Matrix: solid/aqueous

SAMPLE ID

_Surrogate_standard	ds_not_requi	ired_by_the_me	thod		
				-	
QC Limits* (Air)					

- * QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- * If QC limits are not available, use limits of 80 120 % for aqueous and 70 130 % for solid samples.

Actions:

QUALITY	%R < 10%	%R = 10% - LL	%R > UL
Positive results	J	J	J
Nondetects results	R	UJ	Accept

Surrogate action should be applied:

If one or more surrogate in the VOC fraction is out of specification, but has a recovery of > 10%.

If any one surrogate in a fraction shows < 10 % recovery.

All criteria were met
Criteria were not met
and/or see belowN/A

VII. A MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

This data is generated to determine long term precision and accuracy in the analytical method for various matrices. This data alone cannot be used to evaluate the precision and accuracy of individual samples. If any % R in the MS or MSD falls outside the designated range, the reviewer should determine if there are matrix effects, i.e. LCS data are within the QC limits but MS/MSD data are outside QC limit.

1. MS/MSD Recoveries and Precision Criteria

The laboratory should use one MS and a duplicate analysis of an unspiked field sample if target analytes are expected in the sample. If target analytes are not expected, MS/MSD should be analyzed.

List the %Rs, RPD of the compounds which do not meet the criteria.

Sample ID:	-	_	Matrix/	Level:	<u>-</u>
MS OR MSD	COMPOUND	% R	RPD	QC LIMITS	ACTION
	ot_required_as_part_o			_D-1946;_blank	_spike_used_to_assess_

Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

MS/MSD criteria apply only to the unspiked sample, its dilutions, and the associated MS/MSD samples:

If the % R for the affected compounds were < LL (or 70 %), qualify positive results (J) and nondetects (UJ).

If the % R for the affected compounds were > UL (or 130 %), only qualify positive results (J).

If 25 % or more of all MS/MSD %R were < LL (or 70 %) or if two or more MS/MSD %Rs were < 10%, qualify all positive results (J) and reject nondetects (R).

A separate worksheet should be used for each MS/MSD pair.

QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.

^{*} If QC limits are not available, use limits of 70 – 130 %.

All criteria were met _____ Criteria were not met and/or see below __N/A___

VII. B MATRIX SPIKE/MATRIX SPIKE DUPLICATE

MS/MSD - Unspiked Compounds

It should be noted that Method TO-15 does not specify a MS/MSD criteria for the unspiked compounds in the sample. A %RSD of < 50% has therefore been utilized as professional judgment.

If all target analytes were spiked in the MS/MSD, this review element is not applicable.

List the %RSD of the compounds which do not meet the criteria.

COMPOUND SAMPLE MS CONC. MSD CONC. % RSD ACTIO	Sample ID:			Matrix/Level/Unit		
	COMPOUND		MS CONC.	MSD CONC.	% RSD	ACTION
					* 0.1	
		1			10	
				2. (3)	<u> </u>	
		- 12				
	- 100					·

Actions:

^{*} If the % RSD > 50, qualify the positive result in the unspiked samples as estimated (J).

^{*} If the % RSD is not calculated (NC) due to nondetected value, use professional judgment to qualify the data.

All criteria were met _	Х_
Criteria were not met	
and/or see below	

VIII. LABORATORY CONTROL SAMPLE (LCS) ANALYSIS

This data is generated to determine accuracy of the analytical method for various matrices.

1. LCS Recoveries Criteria

Where LCS spiked with the same analyte at the same concentrations as the MS/MSD? Yes or No. If no make note in data review memo.

List the %R of compounds which do not meet the criteria

	LCS ID	COMPOUND	% R	QC LIMIT
		ke)_analyzed_in_this_data	_package;_recoveries_	and_RPD
within_i	aboratory_contr	ol_limits		:
· · · · · · · · · · · · · · · · · · ·				

- * QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit
- * If QC limits are not available, use limits of 70 130 %.

Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

All analytes in the associated sample results are qualified for the following criteria.

If 25 % of the LCS recoveries were < LL (or 70 %), qualify all positive results (j) and reject nondetects (R).

If two or more LCS were below 10 %, qualify all positive results as (J) and reject nondetects (R).

2. Frequency Criteria:

Where LCS analyzed at the required frequency and for each matrix? <u>Yes</u> or No. If no, the data may be affected. Use professional judgment to determine the severity of the effect and qualify data accordingly. Discuss any actions below and list the samples affected.

		All criteria were metX Criteria were not met and/or see below		
IX.	FIELD/LABORATORY DUPLICATE PRECISION			
	Sample ID_LCS/LCSD_(laboratory_duplicate)	Matrix:Air		

Field/laboratory duplicates samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than laboratory duplicates which only laboratory performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field duplicate samples.

The project QAPP should be reviewed for project-specific information.

Suggested criteria: RPD ± 25% for air samples. If both samples and duplicate are <5 SQL, the RPD criteria is doubled.

COMPOUND	SQL	SAMPLE CONC.	DUPLICATE CONC.	RPD	ACTION
RPD for lab	oratory o	duplicate (LC	S/LCSD) within	laborati	ory control limits.

Actions:

Qualify as estimated positive results (J) and nondetects (UJ) for the compound that exceeded the above criteria. For organics, only the sample and duplicate will be qualified.

If an RPD cannot be calculated because one or both of the sample results is not detected, the following actions apply:

If one sample result is not detected and the other is greater than 5x the SQL qualify (J/UJ).

If one sample value is not detected and the other is greater than 5x the SQL and the SQLs for the sample and duplicate are significantly different, use professional judgment to determine if qualification is appropriate.

If one sample value is not detected and the other is less than 5x, use professional judgment to determine if qualification is appropriate.

If both sample and duplicate results are not detected, no action is needed.

All criteria were met _	_N/A	
Criteria were not met		
and/or see below	_	

X. INTERNAL STANDARD PERFORMANCE

The assessment of the internal standard (IS) parameter is used to assist the data reviewer in determining the condition of the analytical instrumentation.

List the internal standard area of samples which do not meet the criteria.

- * Area of +40% or -40% of the IS area in the associated calibration standard.
- * Retention time (RT) within \pm 0.06 seconds of the IS area in the associated calibration standard.

DATE	SAMPLE ID	15 001	IS AREA	RANGE	
	andard_not_require			antified_by_external_standard_	
					-
					_
Actions:			3000		_

1. IS actions should be applied to the compound quantitated with the out-of-control ISs

QUALITY	IS AREA < -40%	IS AREA > + 40%
Positive results	J	J
Nondetected results	R	ACCEPT

If a IS retention time varies more than 0.330 seconds, the chromatographic profile for that sample must be examined to determine if any false positive or negative exists. For shifts of a large magnitude, the reviewer may consider partial or total rejection of the data for the sample fraction.

All criteria were met _	X_	_
Criteria were not met		
and/or see below	_	

XII. SAMPLE QUANTITATION

The sample quantitation evaluation is to verify laboratory quantitation results. In the space below, please show a minimum of one sample calculation:

1605308CR1-02A

Methane

RF=

[] = (44180)/(226379851)

= 0.000195 % OK

Criteria were not mel	
and/or see below	_

XII. QUANTITATION LIMITS

A. Dilution performed

SAMPLE ID	DILUTION FACTOR	REASONS FOR DILUTION
All samples dil	uted by a factor of less th	an 2.31
		ALCOHOL: A COLUMN AND A COLUMN
	100	1
	10000	
STATE OF THE PARTY		
Sec.		

3.	Percent Solids		
	List samples which have ≤ 50 % solids		
		y-1155	: 0:

Actions:

If the % solids of a soil sample is 10-50%, estimate positive results (J) and nondetects (UJ)

If the % solids of a soil sample is < 10%, estimate positive results (J) and reject nondetects (R)